



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of:

WAKI, MINORU

Serial Number: 09/673,194

Filed: October 12, 2000

For: WATER-BASED PIGMENT DISPERSION, PROCESS FOR  
PREPARING THE SAME AND WATER-BASED INK CONTAINING THE  
SAME

Group Art Unit: 1714

Examiner: Callie E. Shosho

#8  
12/18/02  
12

DECLARATION UNDER 37 CFR 1.132

Honorable Commissioner

Washington, D.C. 20231

RECEIVED  
DEC 10 2002  
TC 1700

Sir,

Minoru Waki, the inventor residing at c/o MIKUNI SHIKISO  
KABUSHIKI KAISHA, 101, Kokubunji, Mikunino-cho, Himeji-shi,  
HYOGO, Japan duly deposes and says:

1. That he graduated from Himeji Institute of Technology,  
Japan in March 1978;

2. That since April 1978 he has been employed in MIKUNI  
SHIKISO KABUSHIKI KAISHA;

3. That since April 1978, he has engaged in research and  
development.

4. That he has read and is familiar with the instant  
application for United States Letters Patent and Office Action thereto  
mailed August 6, 2002; and

5. That he has made experiments in order to prove the fact

that a pigment dispersion of the present Claim 4 obtained by using an aqueous polyoxyethylene oligoester acrylate has stability during the passage of time compared with a pigment dispersion of Fryd et al. (USP6,262,152) obtained by using m-tetramethylxylene diisocyanate crosslinker used in Fryd et al..

## EXPERIMENTS

The following experiments prove that a pigment dispersion obtained by using an aqueous polyoxyethylene oligoester acrylate has stability during the passage of time, compared with a pigment dispersion obtained by using m-tetramethylxylene diisocyanate crosslinker.

### EXPERIMENT 1

[Predispersion Step]

The following components were prepared and predispersed by kneading in a butterfly mixer at room temperature for 4 hours to give a mixture.

Component	Amount (Part(s))
Green pigment	35.0
(Pigment green 7 (C.I.74260))	
Styrene-methacrylic acid copolymer	8.5
(styrene / methacrylic acid (weight ratio) = 78 / 22. acid value: 170, number average molecular weight: 85000)	
Diethylene glycol	10.0
Isopropylamine	2.0

Ion-exchanged water

43.5

Then, 110 parts of ion-exchanged water was added to the mixture. These were stirred for 30 minutes, so that the styrene-methacrylic acid copolymer was neutralized and dissolved (neutralization rate: about 130 %).

[Dispersion Step]

The thus predispersed and neutralized mixture was subjected to three-pass treatment in a sand mill which was filled with zirconia beads having a diameter of 0.5 mm in a volume of 80 %. As a result, the green pigment was finely dispersed with the styrene-methacrylic acid copolymer to give a dispersion (pH: 8.2).

[Cross-linking treatment Step]

To the above dispersion having pH of 8.2 was added 5 parts of 100 % aqueous polyoxyethylene oligoester acrylate (difunctionality in a molecule), and these were stirred at 85°C for 8 hours. As a result, the styrene-methacrylic acid copolymer in the dispersion was cross-linked (gel percent of cross-linked styrene-methacrylic acid copolymer: 60 %, number average molecular weight of cross-linked styrene-methacrylic acid copolymer: 700000). Then, the cross-linked styrene-methacrylic acid copolymer was strongly adsorbed on the green pigment. The pH of the dispersion at finishing cross-linking reaction was 7.5.

[Adjust Step of pH]

To the dispersion containing the green pigment and the

cross-linked styrene-methacrylic acid copolymer strongly adsorbed thereon was added triethanolamine in an amount of 0.3 % by weight, so that pH of the dispersion was adjusted to 9.1.

The dispersion was subjected to centrifugal separation at 25000 G for 5 minutes and coarse large particles were removed. As a result, a green water-based pigment dispersion having solid matter concentration of 20 % by weight was prepared.

The average particle size of pigment in the green water-based pigment dispersion was 117 nm and viscosity was 4.31 cps.

After the pigment dispersion was allowed to stand in thermostated room at 60°C for 5 days, the average particle size of pigment in the pigment dispersion was 116 nm and viscosity was 4.28 cps.

#### COMPARATIVE EXPERIMENT 1

A green water-based pigment dispersion was prepared in the same manner as in EXPERIMENT 1 except that m-tetramethylxylene diisocyanate crosslinker and 1 drop of 100 % solution dibutyl dilaurate were used instead of the aqueous polyoxyethylene oligoester acrylate.

The average particle size of pigment in the green water-based pigment dispersion was 113 nm and viscosity was 4.50 cps.

After the pigment dispersion was allowed to stand in thermostated room at 60°C for 5 days, the average particle size of pigment in the pigment dispersion was 130 nm and viscosity was 5.12 cps.

#### DISCUSSION

In Experiment 1 wherein an aqueous polyoxyethylene oligoester acrylate was used as a cross-linking agent, the average particle size of pigment in the pigment dispersion and viscosity were almost same before and after the pigment dispersion was allowed to stand in a thermostated room at 60°C for 5 days.

In Comparative Experiment 1 wherein m-tetramethylxylene diisocyanate crosslinker was used as a cross-linking agent, the average particle size of pigment in the pigment dispersion and viscosity increased after the pigment dispersion was allowed to stand in a thermostated room at 60°C for 5 days.

The results show that a pigment dispersion of the present Claim 4 obtained by using an aqueous polyoxyethylene oligoester acrylate has excellent stability during the passage of time compared with a pigment dispersion of Fryd et al. obtained by using m-tetramethylxylene diisocyanate crosslinker.

Therefore, the excellent effect of the instant invention is not obvious from Fryd et al..

The undersigned declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

This 29th day of November, 2002

by Minoru Waki

Minoru Waki

We, the undersigned witnesses, hereby acknowledge that Minoru Waki is personally known to us and did execute the foregoing Declaration in our presence on:

Date: November 29, 2002

Witness

H. Zama

Date: November 29, 2002

Witness

Kurum Tanaka